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IS 8446 (1991): Pesticide - Carbendazim (MBC) WP [FAD 1: Pesticides and Pesticides Residue Analysis]

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(पहला पुनरीक्षण)

Indian Standard

PESTICIDE — CARBENDAZIM (MBC) WP — SPECIFICATION

(First Revision)

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

June 1991

Price **Group** 2

Pesticides Sectional Committee, FAD 1

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Pesticides Sectional Committee had been approved by the Food and Agriculture Division Council.

Carbendazim (MBC) WP are largely used as fungicides in agriculture and horticulture. The material is also used for ‘cane-dips’.

Carbendazim (MBC) WP formulations are generally manufactured to contain 50 percent (m/m) of carbendazim.

This standard was published in 1977 and subsequently four amendments had been issued. This first revision has been taken up to update the various requirements in light of the experience gained on application by the industry and various other interests.

In the preparation of this standard, due consideration has been given to the provisions of the *Insecticides Act, 1968* and Rules framed thereunder. However, this standard is subject to the restrictions imposed under these, wherever applicable.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 ‘Rules for rounding off numerical values (*revised*)’. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

PESTICIDE — CARBENDAZIM (MBC) WP — SPECIFICATION

(First Revision)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for carbendazim (MBC) WP.

2 REFERENCES

The Indian Standards listed at Annex A are necessary adjuncts to this standard.

3 REQUIREMENTS

3.1 Description

The material shall be in the form of a homogeneous powder which shall wet readily on mixing with water, providing suspension suitable for use as a spray.

3.2 Carbendazim (MBC), technical, employed in the formulation of the water dispersible powder concentrate shall conform to IS **8445 : 1991.**

3.3 The material shall also comply with the requirements given in Table 1.

3.3.1 When determined by the method prescribed in **B-1,** the observed carbendazim content, percent (m/m) of any of the samples shall not differ from the declared nominal value by more than tolerance limits indicated below:

Nominal Value, Percent	*Tolerance Limit,* Percent
Up to 9	+10 — 5
Above 9 and below 50	± 5 of the nominal value
50 and above	+ 5 - 3

4 PACKING

The material shall be packed as per requirements given in IS 8190 (Part 1) : 1988.

5 MARKING

5.1 The containers shall bear legibly and indelibly the following information in addition to the information as is necessary under the ***Insecticides Act, 1968*** and the Rules :

a) Name of the material;
b) Indication of the source of manufacture;
c) Date of manufacture and date of expiry;
d) Batch number;
c) Net mass of contents;
f) Nominal carbendazim (MBC) content, percent (m/m); and
g) The cautionary notice worded as in ***Insecticides Act, 1968*** and the Rules.

Table 1 Requirements for Carbendazim (MBC) WP

(Clause 3.3)

Sl No.	Characteristic	Requirement	Method of Test, Ref to: Annex of This Standard	Method of Test, Ref to: Cl No. of IS 6940 : 1982
(1)	(2)	(3)	(4)	(5)
i)	Carbendazim (MBC) content, percent by mass	Nominal value as declared on the container (see 3.3.1 and 7.2)	B	—
ii)	Material passing through 75 micron IS Sieve [see IS 460 (Part 1) : 1985] after accelerated storage, percent by mass, *Min*	99’0	—	11.1
iii)	Surpensibility, after accelerated storage, percent by mass, *Min (see* Note)	60·0	C	—
iv)	Acidity (as H_2SO_4), percent by mass, Max	0’05	—	11.3.2
	or			
	Alkalinity (as NaOH), percent by mass, *Max*	0’05	—	11.3.3

NOTE — The material shall not be subjected to accelerated storage treatment if it has crossed half of its shelf-life as ascertained from its dates of manufacture and expiry declared on the container.

5.2 Each container may also be marked with Standard Mark.

6 SAMPLING

Representative samples of the material shall be drawn as prescribed in IS 10627 : 1983.

7 TESTS

7.1 Tests shall be carried out by the methods referred to in col 4 and 5 of Table 1.

7.2 **Criteria for Conformity (Bulk Sampling)**

When material is offered in bulk and sampled according to the provisions given in IS 10627 : 1983, the average carbendazim (MBC) content in samples, not more than 90 days old, shall not be less than the declared nominal value.

7.3 **Quality of Reagents**

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070 : 1977) shall be employed in tests.

NOTE--'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A

(*Clause 2*)

LIST OF REFERRED INDIAN STANDARDS

IS No.	*Title*
460 (Part 1) : 1985	Specification for Fitness: Part 1 Wire cloth test sieves (*third revision*)
1070 : 1977	Water for general laboratory use (*second revision*)
6940 : 1982	Methods of test for pesticides and their formulations (*first revision*)
8190 (Part 1) : 1988	Requirement of packing of pesticides : Part 1 Solid pesticides (*second revision*)
8445 : 199 1	Carbendazim (MBC), techical (*first revision*)
10627 : 1983	Methods for sampling of pesticidal formulations

ANNEX B

[*Table* 1, *Item* (i)]

DETERMINATION OF CARBENDAZIM (MBC) CONTENT

B-l PRINCIPLE

B-l.1 Carbendazim is a weak base due to the tertiary nitrogen atom in the benzimidazole ring and can be titrated with perchloric acid in acetic acid in non-aqueous medium.

B-2 REAGENTS

B-2.1 *Acetic Acid* — glacial, anhydrous.

B-2.2. *Acetic Anhydride*

B-2.3 Crystal Violet Indicator — *0'5* percent *(m/v)* in acetic acid.

B-2.4 Perchloric Acid — 0'1 N, freshly prepared by dissolving 8'5 ml of 70 percent perchloric acid in glacial acetic acid. Add 15 ml of acetic anhydride cautiously, make up to 1 litre and keep overnight.

B-3 PROCEDURE

B-3.1 Weigh accurately about 1 g of the sample and transfer into a 60-ml centrifuge tube. Add 25 ml of distilled water. Stir well. Rinse the glass rod with 15 ml of distilled water. Centrifuge the suspension at 2 500 rotations per minute for 30 minutes. Decant the supernatant liquid without disturbing the sediment. Transfer the sediment quantitatively with 25 ml of glacial acetic acid into a clean dry conical flask. Rinse the centrifuge tube with two 15 ml amounts of glacial acetic acid. Add 10 ml of acetic anhydride to a conical flask. Warm the contents at 50-70°C on a water bath for 5 minutes. Cool to room temperature. Filter the contents under suction through a sintered glass crucible into a dry flask. Rinse the crucible with three 10 ml portions of glacial acetic acid. Discard the residue and titrate the filtrate against 0’1 N perchloric-acid using crystal violet indicator. The end point is from blue to green.

B-3.2 Carry out a blank determination using the method given under B-3.1.

B-4 CALCULATION

$$\text{Carbendazim (MBC) content percent by mass} = \frac{19{\cdot}1 \times N \times (V_1 - V_2)}{M}$$

where

N=normality of perchloric acid;

V_1=volume, in ml, of standard perchloric acid required for the sample,

V_2=volume, in ml, of standard perchloric acid required for the blank determination, and

M=mass, in g, of the sample.

B-5 STANDARDIZATION OF PERCHLORIC ACID

B-5.1 Procedure

B-5.1.1 Weigh accurately about 0’2 g of potassium hydrogen phthalate and dissolve in 50 ml glacial acetic acid by warming for a short time. Titrate the solutions with 0’1 N perchloric acid with two drops of crystal violet as indicator, the end point being blue to green.

B-5.1.2 Carry out blank determination with 50 ml of glacial acetic acid and two drops of crystal violet.

B-5.2 Calculation

$$\text{Normality} = \frac{4'898 \times M}{V_1 - V_a}$$

where

M=mass, in g, of potassium hydrogen phthalate;

V_1=volume, in ml, of perchloric acid required for potassium hydrogen phthalate (see B-5.1.1); and

V_2=volume in ml of perchloric acid required for blank determination (see B-5.1.2)

ANNEX C’

[*Table* 1, *Item* (iii)]

DETERMINATION OF SUSPENSIBILITY AFTER ACCELERATED STORAGE

C-l METHOD

C-l.1 Accelerated Storage

Carry out accelerated storage test as prescribed *in* 11.1.2.1 of IS 6940 : 1982.

C-l.2 Sospensibility Test

Weigh accurately 5 g of the treated material (see C-l.1) into 100-ml beaker. Add to it 30 ml of standard hard water (see II.2.2.1 of IS 6940 : 1982) at 30 ± 1°C, work the mass into a uniform slurry and allow it to stand for 15 minutes at 30± 1°C. Transfer the slurry to a graduated cylinder. Wash any residue in the beaker with small quantities of standard hard water at 30± 1°C. Transfer the washings to the cylinder. Fill the cylinder to the 250 ml mark with standard hard water at 30± 1°C. Close the cylinder with the stopper and invert it sharply through 30 complete cycles within one minute, thermally insulating the cylinder from the hands to maintain the temperature of the contents at 30± 1°C during the operation. Allow the cylinder to stand at rest for 30 minutes in a water bath maintained at 30 ± 1°C. (The bench on which the cylinder is placed requires to be protected from vibrations.) At the end of the settling period (30 miuntes), dip the nozzle of the glass tube into the supernatant liquid contained in the cylinder and withdraw nine-tenths (225 ml) of the suspension within 10 to 15 seconds using the vacuum pump. During its withdrawal, the suspension should be disturbed as little as possible, which could be achieved by maintaining the nozzle of the glass tube just below the sinking top level of the suspension. Discard the suspension so withdrawn.

NOTE — Should excessive flocculation occur during the test, the material shall be deemed unsatisfactory. Excessive flocculation in this context means either curdling up of the suspension in the entire column or the appearance of the transparent liquid accompanied by total or partial curdling up.

C-l.3 Transfer the retained one-tenth of the suspension including the sediment into two 60 ml centrifuge tubes. Rinse the cylinder with two 20-ml portions of distilled water and transfer them quantitative into the centrifuge tubes. Centrifuge the suspension at 2 500 rotation per minute for 30 minutes. Decant the supernatant liquid without disturbing the sediment. Transfer the sediment of both the tubes into a dry conical flask using 25 ml glacial acetic acid for each tube. Rinse each of the centrifuge tube with two 15-ml portions of glacial-acetic acid and transfer them to the same conical flask. Add 10 ml of acetic anhydride and warm the contents at 50-70°C in a water bath for 5 minutes. Cool to room temperature. Filter the contents under suction through a sintered glass crucible into a dry flask. Rinse the crucible with three 10-ml portions of glacial acetic acid. Discard the residue and titrate the filtrate against 0·1 normal perchloric acid using crystal violet indicator by the same method as has been employed for determination of pesticide content.

C-2 CALCULATION

C-2.1 Suspensibility after accelerated storage, percent by mass $= \dfrac{1000\ (M-m)}{9M}$

where

M = mass, in g, of the carbendazim present in the treated material taken for the preparation of the suspension (see C-1.2), and

m = mass, in g, of the carbendazim found in the suspension including sediment remaining in the graduated cylinder (see C-1.2).

Standard Mark

The use of the Standard Mark is governed by the provisions of the ***Bureau of Indian Standards Act, 1986*** and the Rules and Regulations made thereunder. The Standard Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well defined system of inspection, testing and quality control which is devised and supervised by BIS and operated by the producer. Standard marked products are also continuously checked by BIS for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

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AMENDMENT NO. 1 MAY 1994
TO
IS 8446 : 1991 SPECIFICATION FOR CARBENDAZIM (MBC) WP

(***First Revision***)

(*Page* **1,** *Table* 1):

a) *Sl* No. (ii), *col* 2 }
b) *Sl* No. (iii), *col* 2 } — Delete the words ‘after accelerated storage’.

(*Page* **2,** *clause* **6**) — Substitute the following for the existing:

‘When freshly manufactured material in bulk quantity is offered for inspection, representative samples of the material shall be drawn and tested as prescribed in IS 10627 : 1953 within 90 days of its manufacture. When the material is offered for inspection after 90 days of its manufacture, sampling shall be done as prescribed in IS 10627 : 1983. However, the criteria for conformity of the material when tested, shall be the limits of tolerances, as applicable over the declared nominal value and given under clause 3.3.1 of the standard.’

(FAD 1)

Reprography Unit. BIS, New Delhi, India

AMENDMENT NO. 2 MARCH- 1996
TO
IS 8446 : 1991 PESTICIDE — CARBENDAZIM (MBC) WP — SPECIFICATION

(First Revision)

(*Page* 2, *clause* 7.3) — Substitute 'IS 1070 : 1992' *for* 'IS 1070 : 1977'.

(*Page* 2, *Annex* A) — Substitute 'IS 1070 : 1992 Reagent grade water (*third revision*)' *for* '1070 : 1977 Water for general laboratory use (*second* revision)'.

(*Page* 2, *Annex* B) — Substitute the following for the existing:

'ANNEX B

DETERMINATION OF CARBENDAZIM (MBC) CONTENT

[*Table* 1, *Item* (i)]

B-l GENERAL

Either of the two methods, namely, High Performance Liquid Chromatography (HPLC) method (see B-2) or titration method (see B-3) shall be used for the determination of carbendazim content. However, HPLC method shall be the referee method.

B-2 BPLC METHOD

B-2.1 Principle

The sample is dissolved in methanol and sulphuric acid along with preliminary solvent mixture and the active ingredient content is determined at 281 nm by comparing the peak areas of carbendazim standard and sample.

B-2.2 Apparatus

High Performance Liquid Chromatograph — Equipped with a printer-plotter-cum-integrator and ultra-violet (UV) detector. The suggestive operating conditions are given below and may be varied depending upon available instrument provided standardization is done:

Column —	Stainless steel, C 18, 250 to 300 mm length and 4.0 to 4.6 mm ID.

Gr 1

Mobile Phase — Mixture of distilled water, methanol and 0.5 M (1 N) sulphuric acid in glass distilled water, in the ratio of 65:35:0.5 (*v/v/v*).

Detector — **UV at 281 nm.**

Flow Rate — 1 ml/minute (approx).

B-2.2.1 ***Microbe Syringe — 20 µl.***

B-2.2.2 ***Volumetric Flask*** — 100 ml.

B-2.2.3 ***Pipette*** — 10 ml.

B-2.3 **Reagents**

B-2.3.1 ***Sulphuric*** **Acid** — 1 N.

B-2.3.2 1, 4 —dioxane -- HPLC Grade.

B-2.3.3 Methanol — HPLC Grade.

B-2.3.4 ***Whatman Filter Paper No. 42***

B-2.33 Glass Distilled Water

B-2.3.6 Preliminary Solvent Mixture — A mixture of *60* parts of **1,4-dioxane** and 40 parts (v/v) of 1 N **Sulphuric** acid.

B-2.3.7 ***Carbendazim Reference Standard*** — of known purity.

B-2.4 Procedure

B-2.4.1 ***Preparation of Reference Standard Solution***

Weigh 0.5 g of carbendazim reference standard in a **100-ml** volumetric **flask** and make up the volume using the **preliminary** solvent mixture (see B-2.3.6). **Shake** tbe volumetric flask vigorously to ensure complete dissolution. Take out 10 ml of this solution with a pipette into another **100-ml** volumetric flask **and make** up **the** volume using the mobile phase.

B-2.4.2 ***Preparation of Sample Solution***

Weigh accurately a quantity of sample equivalent to 0.5 g of active ingredient in a **100-ml** volumetric flask and make up the volume using the preliminary solvent mixture (see B-2.3.6). Shake the volumetric flask to ensure complete dissolution. Filter the solution through **Whatman Filter** Paper No. 42 or equivakmt. Take out 10 **ml** of the filtrate into another **100-ml** volumetric flask and make up the volume using the mobile phase.

B-2.4.3 *Estimation*

B-2.4.3.1 Inject 20 µl of the reference standard solution until the peak area of *2* successive injections agree to within *2* percent. Subsequently, inject the standard and sample solutions in succession according to the following sequence:

$R\; S_1\; S_1\; R\; S_2\; S_2\; R\; S_3\; S_3\; R$ . . . .

where

R = reference standard solution injections, and

S_1, S_2, S_3 . . . . = different sample solution injections.

B-2.4.3.2 *Calculation*

$$\text{Carbendazim content} = \frac{\text{Peak area of sample} \times \text{Mass of reference standard} \times \text{Percent purity of reference standard}}{\text{Peak area of reference standard} \times \text{Mass of sample}}$$

NOTES

1 It is advisable to use a Guard Column in series with the main column, of the same stationery phase, which increases the life of the main column.

2 Determine the carbendazim content by taking average of 2 injections.

H-3 TITRATION METHOD

H-3.1 **Principle**

Carbendazim is a weak base due to the tertiary nitrogen atom in the benzimidazole ring and can be titrated with perchloric acid in acetic acid in non-aqueous medium.

B-3.2 Reagents

B-3.2.1 *Acetic Acid* — glacial, anhydrous.

B-3.2.2 *Acetic Anhydride*

B-3.2.3 *Crystal Violet Indicator* — *0.5* percent (m/v) in acetic acid.

H-3.2.4 *Perchloric Acid* — 0.1 N, freshly prepared by dissolving 8.5 ml of 70 percent perchloric acid in glacial acetic acid. Add 15 ml of acetic anhydride cautiously, make up to 1 litre and keep overnight.

B-3.3 Procedure

B-3.3.1 Weigh 1 g of the sample and transfer into a 60-ml centrifuge tube. Add 25 ml of distilled water. Stir well. Rinse the glass rod with 15 ml of distilled water. Centrifuge the suspension at 2 500 RPM for 30 minutes. Decant the supernatant liquid without disturbing the sediment. Transfer the sediment quantitatively with 25 ml of glacial acetic acid into a clean dry conical flask. Rinse the centrifuge tube twice with 15 ml of glacial acetic acid. Add 10 ml of acetic anhydride to *a* conical flask Warm the contents at 50-70°C on a water bath for 5 minutes. Cool to room temperature. Filter the contents under suction through a sintered glass crucible into a dry flask. Rinse the crucible thrice with 10-ml of glacial acetic acid. Discard the residue and titrate the filtrate against 0.1 N perchloric-acid using crystal violet indicator. The end point is from blue to green.

B-3.3.2 Carry out a blank determination using the method given under B-3.3.1.

B-3.3.3 Calculation

$$\text{Carbendazim (MBC) content, percent by mass} = \frac{19.1 \times N \times (V_1 - V_2)}{M}$$

where

N = normality of perchloric acid;

V_1 = volume, in ml, of standard perchloric acid required for sample;

V_2 = volume, in ml, of standard perchloric acid required for the blank determination; and

M = mass, in g, of the sample.

B-3.4 Standardization of Perchloric Acid

B-3.4.1 Procedure

B-3.4.1.1 Weigh 0.2 g of potassium hydrogen phthalate and dissolve in 50 ml glacial acetic acid by warming for a short time. Titrate the solutions with 0.1 N perchloric acid with two drops of crystal violet as indicator, the end point being blue to green.

B-3-4.1.2 Carry out blank determination with 50 ml of glacial acetic acid and two drops of crystal violet.

B-3.4.13 *Calculation*

$$\text{Normality} = \frac{4.898 \times M}{V_1 - V_2}$$

where

M = mass, in g, of potassium hydrogen pbthalate;

V_1 = volume, in ml, of perchloric acid required for potassium hydrogen pbthalate (see B-3.4.1.1); and

V_2 = volume in ml of perchloric acid required for blank determination (see B-3.4.1.2).'

(*Page* 3, Annex C, *Title*) — Delete 'AFTER ACCELERATED STORAGE'.

(*Page* 3, *clause* C-l.1) — Delete and renumber the subsequent clauses.

(FAD 1)

Reprography Unit, BIS, New Delhi, India